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## Effect of fines on the properties of fibre networks

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### ABSTRACT

The fines of mechanical and chemical pulps have a character very distinct from their respective fibre fractions. The fines can be regarded as a special furnish component and the optimum quality and percentage of fines in the sheet depends on the properties of the fibre fraction used. An addition of suitable fines can improve remarkably the properties of a printing paper.

This paper deals with the structural, optical and strength properties of fibre networks, as influenced by fibre coarseness and the introduction of different types of fines. The response of these networks to calendering is also studied. Fibre fractions of different coarseness and lignin content are used. Confocal laser scanning microscopy (CLSM) is used to determine the changes in the structure and microscopic roughness of the sheet.

### INTRODUCTION

The fines of mechanical and chemical pulps have a character very distinct from their respective fibre fractions. The importance of fibre properties has long been considered essential to the paper properties, and research has been focused on modifications, made chemically or mechanically to this fraction. The role of fines in the development of paper properties has received much less attention. In mechanical pulps the fines fraction has been considered of central importance but the potential of fines of chemical pulps has not been fully realized. In either case the possibility of actively controlling the quality and amount of fines in the furnish has been hardly discussed, not to mention the utilisation of fines as a single, special pulp type to give unique sheet properties.

In this presentation parts of the essential literature are referred to and the possibilities of utilizing more extensively the special characteristics of fines in printing papers is discussed. Results obtained from laboratory experiments are presented.

Fines of different types have been added to fibres of different origin and coarseness. The results show that a fines addition gives remarkable improvements to some paper properties. Adding fines offers an interesting possibility as a potential control variable in papermaking.

### **PROPERTIES OF FINES MATERIAL AND FIBRE FRACTIONS**

### The definition of fines

Fines are generally defined as the fraction of pulp that passes through the 200 mesh screen of a Bauer-McNett classifier. Screens with lower wire densities have also been used in separating fines (<u>1,2</u>). Particles of this fraction are appreciably smaller than those of the fibre fractions, generally being smaller than 200  $\mu$ m (<u>3</u>). The smallest particles are of fibrillar nature and have widths in the range of 0.02-0.5  $\mu$ m (<u>4,5</u>) with the average longest linear dimension around 20  $\mu$ m (<u>6</u>) and an equivalent spherical diameter around 10-20  $\mu$ m (<u>7</u>). The term crill has also been used synonymously with the term fines, but, in fact, denotes material that is of fibrillar origin and has been separated with a special instrument designed for this purpose (3,8).

#### Fines of mechanical and chemical pulp

Mechanical pulps contain a large amount of fines, 20 - 35 % by weight. These fines have a special character that gives printing paper the unique combination of high opacity and reasonable strength. The fines have a large surface area that increases the Campbell forces during consolidation of the sheet and improves bonding and interaction between fibres. However, a fairly large proportion of the surface area remains unbonded which increases the light scattering coefficient and opacity of the paper. These features make it possible to increase simultaneously the tensile strength and opacity of the paper. Because the fines increase the interaction between fibres. which are the main load bearing elements in paper, their effect depends on the amount and quality of the fibre fraction. The possibility of improving the papermaking properties of mechanical pulps by increasing simultaneously the amount of long fibre and fines fractions by reducing the middle fraction has been pointed out by Law and Garceau (9) and Lindholm (10). An increase in the amount of fines of mechanical pulps strongly impairs drainage properties of the furnish but improves wet web strength (11). It also affects the properties of paper by increasing apparent density, tensile and burst strength, tear strength (depending on fibre properties), folding endurance, Scott Bond, light scattering and absorption coefficients while strongly decreasing air permeability (1,7,12,13).

A chemical pulp contains a smaller proportion of fines than a mechanical pulp, the percentages, in a virgin pulp, rising from a few percent to 10- 12%, depending on the beating level (<u>14</u>). Fines of chemical pulps have similar effects on pulp and sheet properties to those of mechanical pulps. However they have a rather limited ability to enhance optical properties, but have a strong influence on the bonding degree of the sheet. A better bonding ability results in higher apparent density, tensile strength, elastic modulus, compression strength, rupture elongation and elongation capacity, but somewhat lower light scattering

coefficient (4,5,7,8,14-21,). However some contradictory statements on the importance of fines have also been expressed (8,22).

### **Classifications of fines**

In addition to the difference between fines of mechanical and chemical pulps, other more subtle differences have also been reported. Brecht *et al.*(<u>23</u>) introduced a characterisation for two different types of fines within a mechanical pulp. They called these "**Mehlstoff** (flour stuff)" and **Schleimstoff** (slime stuff) ". The former consists of chunky lignin-rich particles with low bondability, and the latter of cellulose-rich finer and more fibrillar particles with high bondability. The Schleimstoff has been further divided into fibrillar and lamellar fractions (<u>24</u>). Mehlstoff contains a dusty fines fraction which consists of lignin rich particles originating from the middle lamella.

The terms **primary fines** and **secondary fines** are established to distinguish between the fines that are present already in the pulp before refining, like ray and parenchyma cells, piths etc., and those that are created by mechanical actions during beating. The secondary fines contain more fibrillated and lamellar material originating mainly from the S<sub>1</sub>- and S<sub>2</sub>-layers. Secondary fines are more advantageous to paper properties, and have clearly better bonding properties. Secondary fines have been found to increase, and the primary fines to decrease, the specific bond strength (25).

Drying of chemical pulps is known to reduce the reswelling ability of the fibres, an effect called hornification. The same effect takes place also with fines (<u>17</u>). This has some bearing on recycled furnish. The WRV (water retention value) of fines decreases with drying similarly to that of the fibre fraction. Also the fines created during beating of once dried pulps have lower WRV than fines created during beating of never-dried pulps. When the recycling is done on a laboratory scale the deterioration in fines properties is not however drastic (<u>7</u>). The fines from recycled kraft pulps are nearly as valuable as those of virgin kraft pulps in improving the strength properties of handsheets.

### Effects of the mechanical processing on the fines

The fines characteristics also depend on the type of mechanical process the pulp has been subjected to. When the fines of a thermomechanical pulp (TMP), pressure groundwood. (PGW) and conventional stone groundwood (SGW) pulp were compared, the SGW fines had the lowest bonding potential, while the TMP fines (<u>26</u>) or PGW fines (<u>20</u>) had the highest. The bonding potential of fines increased with decreasing freeness of the pulp (<u>26</u>). According to Heikkurinen and Hattula (<u>27</u>) TMP fines consist of material progressively removed from the fibre surfaces. The lignin content of the fines decreases, and the length of particles increases with the amount of energy used in refining.

The production of fines during beating of chemical pulp has been considered to be an effect of higher importance than external fibrillation  $(\underline{14,20})$  for paper strength. Some differences in the quality and quantity of fines created during different beating processes have also been found by Kibblewhite ( $\underline{22}$ ), although differences in their strengthening abilities were not reported. The PFI mill, in contrast to the Lampèn mill and Valley beater, proved to be very effective in progressively removing material from fibre surfaces ( $\underline{28}$ ). Kibblewhite also constructed a fines index based on the extent to which different fibre layers are exposed during beating. A fairly linear correlation was found between the fines index and amount of beating ( $\underline{22}$ ).

### Fines and fibre properties

Paavilainen (<u>14</u>) has reported differences in the amount and quality of fines created during the beating of springwood and summerwood fractions of kraft pulp. Stiffer summerwood fibres are more likely to fibrillate, create fines and break during beating than the more flexible springwood fibres. Summerwood fibres also tend to form somewhat coarser fines than springwood fibres. External fibrillation has a causal relationship with the creation of secondary fines. Stiffer fibres are more in need of fines (<u>14</u>) and can better utilise (<u>7,21</u>) the fines addition to form stronger sheets than more flexible fibres. The fine material is assumed to play an important role also in pulp blends of stiffer

mechanical and more flexible chemical pulp fibres (<u>29</u>). The wood species affects the quality of the fines fraction. Hardwood pulps contain larger proportions of primary fines than softwoods. Mechanical pulps of hardwoods contain more of the "Mehlstoff"-type fines, which do not contribute much to Campbell forces (<u>1</u>). Their quality, however, can be greatly improved by chemical pretreatment.

### The strengthening effect of fines

The fines have been considered to have greatest effect on the fibre bonds. Giertz (18,19) proposes that fines covered with swollen hemicellulose increase the amount of bound water and form a strong wet adhesion system. The high amount of bound water acts as a bondforming substance between fibres. Fines increase Campbell forces and contribute to sheet consolidation, wet web strength and interfibre bonding. The main consequences are increased sheet density and improved activation of fibre segments and fibre wall material. It has been suggested that fines reduce stress concentrations in the bonded regions leading to more uniform stress distributions (30). Nanko and Ohsawa (31) have specified two structural features of a fibre bond where fibrillar fines can contribute to increased bond strength. The 'bonding laver' between two fibre surfaces consists of randomly oriented fibrils of fine material that fill the potential gaps between fibres at the bond areas. The 'covering laver' at the periphery of a fibre bond protects and strengthens the bonding edge which is susceptible to cracking.

### Properties of fines compared to fibre fraction

Several physical and chemical properties have been determined in order to find the essential characteristics of fines. The particle size and the extent to which the material is fibrillated has led to determination of the surface area and particle size of fines. Permeability measurements have shown the fines to have surface areas from 10 to 50 m<sup>2</sup>/g, while the fibre fraction has surface areas in the order of 1 m<sup>2</sup>/g.

Measurement of water retention values (WRV) (5,17), the amount of inaccessible water by the solute exclusion technique (19), sedimentation rate and sedimentation concentration or volume (18) are methods which have been commonly applied to fines fractions. Fines material is rather hydrophilic. The fines of a chemical pulp have a WRV two to three times that of the fibre fraction and the amount of inaccessible water is 5-7 times that of the respective fibre fraction.

The crystallinity of fines has also been measured (<u>4</u>,<u>5</u>). Crystallinities of 64% and 79% were obtained for bleached kraft fines and the fibre fraction, respectively, by Htun and de Ruvo (<u>4</u>) using X-ray diffraction. According to Pryzybysz and Szwarcsztajn (<u>33</u>) the crystallinity of wet primary fines is higher than that of wet secondary fines, while the crystallinity of dry secondary fines is higher. The fines fraction also has a higher water content at certain relative humidity and lower DP than the fibre fraction (<u>4,17</u>).

Little or no differences have been found in the chemical composition of fines and fibre fractions of chemical pulps. The xylan content of fines from a bleached kraft pulp was slightly higher than that of the fibre fraction (<u>4</u>). The primary fines had a higher content of ash and extractives (<u>5</u>). Mechanical pulp fines, especially TMP, have higher lignin content on average than fibres (<u>27,32,33</u>). The fines contain particles originating from the lignin-rich middle lamella and primary wall. The lower the freeness of the pulp the higher is the cellulose content.

The fraction passing through the 200 mesh screen is accompanied by lignin and hemicellulose containing colloidal and soluble material. The amount of material dissolved in defibration and beating depends on the yield of the pulp. With high yield pulps this may amount to several percent of the wood material (<u>34</u>) and may affect the sheet properties. The colloidal hemicelluloses especially have been found to increase the tensile strength and z-strength of paper(<u>35</u>).

### Fines addition as a control variable ?

The amount and quality of fines affects most paper properties. The importance of mechanical pulps has been recognised to be due almost exclusively to the unique properties of its fines fraction (<u>36</u>). The fines deeply affect papermaking processes from the wet end to the dry end altering the functional behaviour of paper. In Table 1 we can see numerous properties, based on published literature, that are affected by the amount of fines. Considering the most important properties required of printing papers, such as optical properties, printability and runnability, we can anticipate that fines additions could make a considerable positive contribution to many of them. This raises the question why not use fines as a special additional raw material type and control its properties independently of the rest of pulp.

There are few instances where the possibility of using fines quality and amount as an active control variable has been explicitly mentioned. Hartman (<u>20</u>) has studied the different primary effects of beating on sheet strength and concluded that the main effects of beating are internal fibrillation of fibres and creation of fines. He states: "Internal fibrillation and fines can be produced in separate steps, indicating that an additive approach to developing sheet properties through refining is possible."

Perekalskiî and Filatenkov (<u>37</u>) and Aaltio (<u>15</u>) have independently studied the possibilities of improving the strength of paper. By adding highly beaten (SR 87), hemicellulose-rich birch pulp to a pine sulphate pulp beaten to SR 23 the strength properties could be greatly increased. The highest strength increases were at 50% addition. The optimal addition seemed to be at a local maximum of around 12.5% to 15%, where the increase in tensile strength was 22%. The addition made it possible to reduce the amount of beating of the pine sulphate pulp thus giving considerable energy savings.

# TABLE I. Effect of fines on the

## pulp and sheet properties

Property	Effect
Drainage resistance	+ +
Wet web strength	+
Sheet density	+
Shrinkage potential	누
Tensile strength	+
Elongation	+
Tensile stiffness	+
Tear strength	-
Compression strength	+
Folding endurance	+
Air permeability	
Specific bond strength	
-primary fines	-
-secondary fines	+
Light scattering	
-chemical pulp fines	(-)
-mechanical pulp fines	+ +
Fibre rising	-
Linting	-

+ the property value increases

- the property value decreases

,

### **OBJECT OF STUDY**

The object of the research was to find out the potential advantages of using fines as a separate furnish component with special regard to the demands commonly made for printing papers. The study aimed at finding the effect of different types of fines on paper structure, functional properties and response to calendering. The study was made on a laboratory scale.

The preliminary results had indicated that in a chemical pulp the long fibre and fines fractions constitute the two most important fractions. The same fact had been shown earlier with mechanical pulps (9,10). For this reason only long fibre and fines fractions were used in the experiments. White water recirculation was used always when the furnish contained fines. Handsheets were wet pressed using 490 kPa wet pressing pressure for 4 min and dried between blotters on a drum drier.

### EFFECT OF FINES TYPE ON NETWORK PROPERTIES

### Addition of TMP and kraft fines to kraft fibres

Fines of a low freeness TMP and that of extensively beaten kraft pulps were added to the long fibre fraction (+20 mesh) of a slighly beaten bleached pine kraft pulp. In Figs.1a and 1b we can see the surfaces of papers containing TMP and kraft fines. The TMP fines contain coarse cell wall material, broken fibres, parenchyma cells, fibril bundles, lamellar particles and bordered pits. In the sheet they behave partly like loosely bonded filler material padding out pores and spaces in the fibre network, creating new light scattering surfaces and open structures. The kraft fines are of a more compacting quality containing not only individual fibrils but also larger lamellar particles and parenchyma cells. In a sheet they tend to cover fibres densely and form membrane-like structures.



Fig. 1a Surface of handsheet containing of 10 % kraft fines (above) and Fig. 1b Surface of handsheet containing 10 % TMP fines (below).



Fig. 2 The effect on tensile strength of adding kraft and TMP fines to kraft fibres.

The tensile strength of the kraft fibre network increased strongly with the addition of kraft fines to the long fibres (Fig. 2). An addition of 15% kraft fines nearly doubles the tensile strength. The improvement is more moderate with a TMP fines addition.

The fines begin to affect the network structure at the formation stage. Drainage is impaired and sheet density is higher (Table 2) probably due to increased Campbell forces. Kraft fines make the sheet denser than TMP fines do. This could be due to chemical differences or due to differences in particle size and resulting specific surface area.

The particle size distributions were measured by laser diffraction and the Coulter method. The hydrodynamic specific volume of the sediments of fines were determined after centrifugation, and the specific surface area of the respective sediments measured after freeze drving. The results can be seen in Table 4. Both types of fines contained surprisingly large particles, up to several hundred micrometers in size. The hydrodynamic specific volumes of the fines were nearly the same. The BET surface area of kraft fines was distinctly higher but also the median particle size of the kraft fines was higher than that of TMP fines. This indicates that, on average, kraft fines consist of larger particles than TMP fines but the particles are more fibrillated. This accords with microscopical observations. In white water the particle size was, of course, distincly smaller than in the original fines slurry but the median equivalent sperical diameter of kraft fines was smaller than that of TMP fines. These results suggest that the higher density of kraft pulp handsheets is partially due to the particle size or specific surface area but is also related to the chemical differences between the fines. The main chemical difference between these fines is the high content of lignin in TMP fines. It causes two potential restrictions: 1) the polarity of lignin is low and so are the surface forces between the lignin containing surfaces and water. 2) the lignin makes the TMP fines stiff in the wet state thus enabling them to resist surface tension forces. The better drainage rate of sheets with TMP fines (Table 3) demonstrates their less strong interaction with water compared with kraft fines.

Furnish	Drainage time, s	Amount of fines in sheet,%	Apparent density, kg/m <sup>3</sup>
Kraft fibres	6	-	440
Kraft fibres+15% TMP fines	10	14.2	494
Kraft fibres+15% kraft fines	28	14.1	571

 Table 2. Effect of fines addition on drainage, retention and sheet

 density

Table 3. Properties of TMP and bleached kraft fines

Type of fines	Median diameter, μm (Laser diffraction)	Median diameter in white water, μm (Coulter method)	Hydrodynamic specific vol., cm <sup>3</sup> /g (5725g, 5 min)	BET adsorption area, m <sup>2</sup> /g
TMP-fines	32.6	16	54	7.9
Kraft fines	61.5	8	56	13.9



Fig. 3. Effect of adding TMP and kraft fines to the long fibre fibre fibre fraction of a bleached kraft pulp. The addition of 1.2% cationic starch before fines addition does not increase the drying stress much.



Fig. 4 The effect of wet pressing, beating and fines addition on the kraft fibre network. The kraft fibres are latewood fibres of high coarseness .

Fines increase the drying stress of handsheets (Fig. 3). The high water content of swollen fines material and increased Campbell forces are believed to be responsible for this. The shrinkage forces arising in restrained drying affect the way the fibres and the fibre network will deform in straining. The fines addition increases the tensile strength and tensile stiffness, rupture elongation, tensile energy absorption as well as Scott Bond or z-directional tensile strength.

### Possible mechanisms causing strength improvement

As mentioned earlier, fines increase sheet strength and density. Some understanding of the mechanisms behind the strength increase can be approached if we compare the effect of fines with other means of increasing the density of the handsheets, such as beating and wet pressing. Such an experiment was accomplished and the fines were removed from the fibre fraction after beating. In Fig. 4 we can see the effect of wet pressing pressure, amount of beating and kraft fines addition. The fibre fraction in this case was that of long kraft fibres of high coarseness. The high density caused by fines addition is capable of increasing the strength of the network as effectively as beating of the fibre fraction, and much more effectively than wet pressing. This suggests that the effect of increased swelling and flexibility of fibres caused by beating has the same kind of effect on the network as the fines addition. Giertz (18, 19) and Lobben (38) have described the effect of beating on the tensile properties as an activation of the network. According to Lobben two kinds of activation takes place. Firstly, beating increases the size and the number of the bonds. The number of seaments between bonds is increased and their length reduced. Secondly, drying under tension results in improved tensile strength and stiffness of fibres or fibre segments. This was first demonstrated by Jentzen (39) with individually dried fibres and more recently by Wuu (40) et al. by testing "cut-out" fibres in machine and cross machine direction. It seems probable that fines function by both of these mechanisms.



Fig. 5. The effect of TMP and kraft fines addition on the NBS-specific bond strength values of a kraft fibre network.

The effect of fines addition on the strength of fibre bonds can be evaluated by the Nordman Bonding Strength method. The Nordman Bonding Strength (NBS) measurement gives a specific bond strength value that is the ratio of the plastic energy of the stress-strain cycle divided by the respective change in light scattering coefficient. Values have been shown to be consistent values regardless of whether the same sheets are dried under restraint or freely (<u>41</u>). According to NBS measurements, beating of a bleached pine kraft pulp does not, after an initial increase, affect the specific bond strength. Drying under restraint gives only a marginally better NBS-value than drying the sheet freely. As an energy based value NBS does not take into account the order of loading of the elements. The fines addition (Fig. 5) seems to have little effect on the NBS value compared with the effect of starch. Kraft fines improve the NBS-value but TMP fines do not.

When we inspected more closely the phenomena taking place in stressstrain cycling experiment we found that the limit where the plastic elongation started did not change by fines or starch addition nor did the relationship between total elongation and nonelastic elongation. This indicates that no qualitative change in the elongation behaviour took place. However the rupture process of fibre bonds was affected. We can evaluate the amount of bond ruptures based on the change of light scattering coefficient. Increasing the kraft fines content increased the stress (Fig. 6) and strain needed to start the bond breakages. An addition of 1.2% starch to the fibres did not necessarily change the starting point of bond breakages but did greatly decrease the rate of bond ruptures. Also the shape of the load elongation curve was changed. The kraft fines addition increased both the primary and secondary slope of the stress-strain curve. Starch addition increased mainly the secondary slope.

The amount of material bearing load at the secondary regime can be estimated from the deloading modulus (or deloading stiffness index) of the stress-strain cycle (Fig. 7). The deloading stiffness indices increase linearly with increased strain, indicating that more material is activated to bear load. The kraft fines addition elevates the curves without affecting the slope. Starch addition does not have much effect at all. Thus, although starch increases the secondary slope of the stress strain curve, it is not due to an increased amount of material bearing the load but due to decreased plastic deformation taking place at bond areas.



Fig. 6. The effect of kraft fines addition and starch addition on the change of light scattering coefficient in a stress strain cycle. Each regression line is based on 30 measurements.



Fig. 7 The effect of kraft fines addition and starch addition on the deloading stiffness index. Each regression line is based on at least 14 measurements.

A kraft fines addition activates the network to bear load from the beginning of straining, and postpones the start of bond ruptures. The rate of bond ruptures is not changed, but the secondary slope is somewhat increased. TMP fines behave qualitatively the same way as kraft fines but less effectively. These strengthening effects of fines additions are not unique. The previously described effects of a fines addition on the straining behaviour of the network are the same as those resulting from an increased amount of beating



Fig. 8 The effect of TMP and kraft fines addition on the tensile index-light scattering coefficient combination.

### Effects of fines additions on strength and optical properties

When strength properties of a chemical pulp are improved, for example by beating, the optical properties are impaired. They form a critical pair of properties. It is thus interesting to inspect the effects of fines addition in this coordinate system shown in Fig. 8. The addition of TMP fines to kraft fibre networks improves the light scattering but does not increase the tensile strength appreciably. The kraft fines behave the other way round: they strongly increase the tensile strength but simultaneously the light scattering coefficient decreases slightly. The properties of the handsheets are thus very strongly dependent on the quality and amount of fines added.

### The quality of fines created during beating

The S<sub>2</sub>-wall of fibres has a high cellulose content. The fibrils produced by this layer might have superior strengthening properties compared with those of the surface layers, as is the case with the fines of a mechanical pulp (26,27). To test this hypothesis the strengthening ability of fines produced in differet stages of beating was measured. A bleached pine kraft pulp was fractionated and the primary fraction (-200 mesh) collected for sheet making. All the other fractions were beaten in a Valley beater for 30 minutes. The fines created during beating were separated. The beating was continued and the procedure repeated so that fines were collected also after total beating times of 60 and 120 minutes. Each of the fines fractions was blended with the long fibre fraction of kraft pulp (beaten 20 min in a Valley beater) giving a fines content of 15 %. Handsheets were made with white water recirculation, wet pressed using 490kPa pressure and dried on a drum between blotters.



Fig. 9 Effect of kraft fines, from different stages of beating, on tensile strength of a kraft fibre network.

The tensile strength of the handsheets containing fines was distincly higher than the strength of the pure fibre fraction (Fig. 9). Also the primary fines were able to improve the tensile strength. The fines created during beating, however, are distinctly better than the primary fines for improving strength. There is no dramatic difference between the fines produced in the different stages of beating. They all have good bondability. The median size of the fines fraction tends to increase with beating while the volume percentage of particles smaller than 10  $\mu$ m remains nearly the same (Table 4).

# Different types of fines; Neutral sulphite (NSAQ) pulp fines and fines produced from kraft pulp swollen by alkaline treatment

There are may be methods to produce fines by a less damaging process than just beating. One possibility is to break the internal coherence of fibres chemically or by a combination of mechanical and chemical action. To test this hypothesis a bleached kraft pulp swollen in sodium hydroxide was beaten in a PFI mill for 80 000 revolutions. Two NaOH concentrations, 5% and 10%, were used. The fines were collected as before.

A high hemicellulose content is commonly thought to improve bonding of fibres and fines. A pulp of high hemicellulose content is made commercially from Norway spruce by a neutral sulphite cooking process with addition of antraquinone (NSAQ-process). The pulping process is followed by a peroxide bleaching. The yield is around 57% (<u>42</u>). The hemicellulose content is over 20% (<u>43</u>). The lignin content is about 6 %. According to Kettunen (<u>43</u>) this pulp has nearly twice the Nordman Bonding Strength of an unbleached kraft pulp. The strengthening ability of NSAQ fines produced during Valley beating of this pulp was studied.

The combined alkaline treatment and beating of the kraft pulp did not produce fines with improved strengthening properties. Their actual strengthening ability was of the same magnitude as that of TMP fines (Fig 10). The higher alkaline concentration gave fines of lower quality probably due to greater extraction of hemicelluloses. However, the fines produced by beating of the NSAQ pulp gave fines of high bonding ability and high tensile strength. Based on these results the physical median particle size seems to be of7 less importance than the chemical composition of the fines.



Fig. 10 The effect of fines of Valley beaten NSAQ pulp and fines of alkaline extracted and PFI beaten kraft pulp on tensile strength of kraft fibre network.

 Table 4. Particle size of fines measured by laser diffraction method.

Source of fines	Median equivalent spherical diameter,	Volume-% <10 μm
	um	
Unbeaten pulp	47.2	7.4
30 min beaten	49.9	7.6
30-60 min beaten	66.8	7.9
60-120 min beating	70.1	8.6
TMP fines	32.6	17.3
Kraft fines (NaOH-10%)	40.0	13.5
NSAQ fines	60.4	4.5



Fig. 11 Effect of TMP and kraft fines addition to the TMP fibre network and blend of TMP/kraft fibres. The TMP/kraft fibre weight ratio in the blend was 55/45.



Fig. 12 By adding a suitable mixture of TMP and kraft fines any point between the two curves can be reached.



Fig 13. The tensile strength and light scattering combination of kraft/TMP fibre network can be affected much more strongly by appropriate fines addition than changing the composition of the fibre fraction.

# INFLUENCE OF ADDING FINES TO FIBRE FRACTIONS OF DIFFERENT PULPS

### Addition of fines to TMP fibres and TMP/kraft fibre blend

The addition of fines to a TMP long fibre fraction (+30 mesh) has rather similar effects on the tensile strength and optical properties as it has on the kraft fibre network (Fig. 11). The TMP fines improve light scattering coefficient strongly and also strengthen the fibre network. The kraft fines improve the tensile strength strongly but the light scattering coefficient is impaired. If we add fines to a blend of TMP fibres and kraft fibres the curve is only a little shifted. The weight percentages of TMP and kraft fibres in the blend were 55% and 45%, respectively. The large number of kraft fibres is hardly able to strengthen the network.

Mixtures of kraft and TMP fines were also added to the fibre blend. It is interesting that, in this case, depending on the amount of kraft fines in

the mixture we could move linearly from the right wing to the left wing of the curve (Fig. 12). It implies that any combination of properties inside the triangle can be achieved by an appropriate fines addition. It can also be deduced from Fig. 13 that an addition of 5-10% kraft fines is a more beneficial way to improve the strength of the TMP/kraft fibre network than is the addition of kraft fibres.

### Addition of fines to earlywood and latewood kraft fibres

Fibre coarseness is probably the most important single fibre property in determining paper properties. The coarseness affects the strength properties and optical properties, the network structure, smoothness and permeability of paper (41, 44, 45). In many cases the low coarseness fibres yield better strength and optical properties. However, coarse fibres result, for example, in a better tear strength and specific bond strength and produce bulkier sheets than low coarseness fibres. The coarse fibres however tend to form rough paper. The smoothness of paper can be improved by increasing the fines content. But can the smoothness of a coarse fibre network be increased to a reasonable level by fines addition? This topic was studied using bleached pine kraft fibres.

# Table 5. Properties of earlywood and latewood kraft fibres and TMP fibres

	Fibre length,	Coarseness,	Curl index*
	mm	mg/m	
Earlywood fibres (+20 mesh)	3.03	0.204	0.161
Latewood fibres (+20 mesh)	2.98	0.324	0.174
TMP fibres (+30 mesh)	2.50	0.307	0.042

\*measurement based on the definition of Jordan and Page ( $\frac{L}{1}$  - 1)

Two fractions were separated by a centrifugal cleaner after multiple recirculations as described in references 41 and 45. The long fibre fractions (+20 mesh) were collected using a Bauer-McNett fractionator. These two long fibre fractions are in the following discussion called earlywood and latewood fractions. Their properties are presented in

Table 5. There was no difference in the weighted length of the earlywood and latewood fibres. The curl index of latewood fibres was somewhat higher than that of the earlywood fibres, but they both were more curled than TMP fibres.



Fig. 14 Effect of fines addition on the tensile strength of earlywood and latewood kraft fibre networks.

The strength, apparent density and tensile strength of the unbeaten latewood fibre handsheets was very low. The latewood fibre fraction was beaten in a PFI mill for 4000 revolutions to make paper of the same density as the unbeaten earlywood fraction (Table 6). The tensile strength of beaten latewood handsheets was higher but the light scattering coefficient and  $\beta$ -formation, expressed as standard deviation, were poorer for the latewood sheets. Roughness and permeability were considerably higher for the earlywood sheets.

Fig. 14 illustrates the effect of fines addition on the tensile strength of earlywood and latewood handsheets. The tensile strength of latewood fibres could be increased by both TMP and kraft fines addition. The addition of 30% kraft fines increased the tensile index of unbeaten and beaten latewood fibre sheets by the same amount (64 Nm/g) in both cases. The response to fines addition of earlywood fibres is stronger than that of latewood fibres. The strength of the earlywood fibre network

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		Drainage time,	Density	Bending stiffn. (L&W)	Tensile index	In-plane tear	Light scatt.	β–formation, Stdev	Roughness (Bendtsen)
Fines fraction	Fibre fraction	Ś	kg/m3	N N N	0/mN	Nm2/kg	m2/kg	g/m2	ml/min
No fines	Earlywood, unbeaten	9	440	40.1	25.5	15.7	37.3	3.8	1630
No fines	Latewood, unbeaten	9	282	22.4	2.3	0.8	23.4	4.2	4602
No fines	Latewood, beaten	9	430	37.1	34.7	13.2	17.4	4.2	4020
TMP fines	Earlywood, unbeaten	10	494	41.4	45.2	21.5	45.2	3.9	1450
TMP fines	Latewood, unbeaten	7	349	35.2	14.0	6.4	30.9	4.1	2570
TMP fines	Latewood, beaten	7	466	36.8	45.2	16.8	26.6	4.0	1490
Kraft fines	Earlywood, unbeaten	28	571	36.9	84.9	23.1	30.4	3.8	1505
Kraft fines	Latewood, unbeaten	80	413	43.0	40.7	21.8	27.3	4.1	1970
Kraft fines	Latewood, beaten	ω,	525	40.3	81.9	28.8	19.1	4.1	1690

increases more steeply with kraft fines addition and surpasses that of beaten latewood fibres. [We cannot follow the curve further because earlywood handsheets containing 30% kraft fines had a drainage time of about 20 min and therefore sheetmaking was impractical.] The strength of earlywood sheets is increased more because the kraft fines are able to make the earlywood sheet denser than latewood sheets. The earlywood sheets drain slowly because the fibres conform under less pressure than latewood fibres and there are a greater number of fibres in the sheet which make the pore sizes (polygon area) smaller.

The available tensile strength and light scattering combinations of earlywood fibres are distinctly more beneficial than those for latewood fibres (Fig. 15). The low light scattering coefficient of latewood fibres can be improved by TMP fines addition. But the good light scattering level of unbeaten earlywood fibres can be achieved only by adding 30 % TMP fines, in which case the tensile strength of beaten latewood sheet is superior to that of earlywood fibres. The properties of earlywood sheets can also be improved by fines addition, but the longer drainage times of earlywood sheets set for the potential fines additions, a limit appreciably lower than for latewood sheets.



Light scattering coefficient, m2/kg

Fig. 15 The effect of fines addition on the tensile strength and light scattering of kraft fibre networks.

# Effects of fibre coarseness and fines addition on the calendering response

The roughness of a printing paper cannot be predicted from the roughness of uncalendered handsheets. The apparent density and structure of handsheets is so severely affected in calendering that most of the paper properties, apart from smoothness and resistance to air permeance, deteriorate. This degree of deterioration depends on the fibre properties and calendering variables such as nip load, roll temperature, moisture content and properties of the rolls.

In this study earlywood and latewood handsheets were calendered using steel rolls in a Ramish laboratory calender and applying increasing nip loads until the desired smoothness level (Bendtsen 100 ml/min or Parker Print Surf 4  $\mu$ m) was reached. The calendering response was evaluated by two different methods. The first evaluation was based on the sheet properties interpolated to a PPS roughness of 4 $\mu$ m and the amount of calendering (expressed as total pressure impulse) needed to reach that level. Secondly the calendering response of the sheets was assessed from microscopical measurements. A confocal laser scanning microscope (Leica CLSM) was used to monitor and measure the changes in areas of 125 $\mu$ m by 125 $\mu$ m as the calendering proceeded. The topography of ten areas were measured before and after each calendering step.

### Effect of calendering on the macroscopic properties of handsheets

The earlywood fibre networks are far easier to calender to the reference smoothness level than latewood fibre networks (Fig. 16). The addition of TMP fines lowers the density level where the desired smoothness is achieved. The reduction is higher for latewood sheets. An addition of 30% fines is not enough to even out the difference between these two types of fibres - a fines content of over 50% would be needed.



Fig. 16 Interpolated density level where Parker Print Surf smoothness 4  $\mu m$  is achieved for handsheets containing TMP fines



Fig. 17 Interpolated density level where Parker Print Surf smoothness 4  $\mu$ m is achieved for handsheets containing kraft fines.

The higher density of beaten latewood sheets indicates that increasing the flexibility of beaten latewood fibres does not make it easier to reach the desired smoothness level. This discrepancy is possibly due to differences in compression behaviour of these fibre networks under the annulus of the smoothness tester.

Addition of kraft fines does not improve the smoothness of the handsheets much and the desired smoothness is reached at only a slightly lower sheet density (Fig. 17). Compared to TMP fines the kraft fines are much less effective in smoothing the fibre networks. This can be attributed to their different behaviour during sheet formation and drying. Kraft fines are flexible and have greater affinity for fibre surfaces. They tend to bond densely to the fibre surfaces they have been drawn to by Campbell forces. TMP fibres are better able to resist Campbell forces and fill the pores and valleys of fibre networks. When the spatial fibre density of handsheets is decreased by increased fibre coarseness and/or yield, the presence of "pore filling" TMP-type fines becomes especially desirable.



Fig. 18 The amount of calendering expressed as total pressure impulse, needed to bring the TMP fines containing hansheets to PPS roughness level 4  $\mu$ m.

The amount of calendering needed to bring the handsheets to the desired smoothness level can be expressed as the total pressure impulse. The initial line loads used were 35 kN/m and the highest were 175 kN/m, two passes on each load level were given to the handsheets. The total impulse given to the latewood sheets was several times that given to the earlywood handsheets in order to reach the desired PPS roughness level (Fig. 18). The addition of TMP fines brings the necessary impulse level down but a large gap between these two fibre types still remains.



Fig. 19 The property combinations for the fines containing handsheets calendered to the PPS roughness level of 4  $\mu m$  .

The higher the amount of calendering the sheets are subjected to, the more the sheet properties are affected. Both tensile strength and light scattering coefficient deteriorate. The properties of latewood fibre handsheets are affected more than those of earlywood fibre sheets. The available property combinations for tensile strength and light scattering can be seen in Fig. 19. The properties of earlywood handsheets have not deteriorated appreciably from the level of uncalendered sheets (see Fig. 15). The properties of latewood handsheets, especially those containing kraft fines, have deteriorated considerably. The difference

between earlywood and latewood handsheets has not diminished on calendering. This difference was reduced only in the case of beaten latewood handsheets with high proportion of TMP fines.

# Effect of fines addition on the microscopical structure of handsheets

The surface structures of handsheets were inspected using a confocal laser scanning microscope (CLSM). It was used to study the paper structure and effect of calendering and fines addition on the microscopic surface structure. It serves as an optical non-contacting profilometer. The basic instrument and the method is described in reference (46). The CLSM enables monitoring of surface details from  $0.2 \,\mu\text{m}$  to  $125 \,\mu\text{m}$ . The z-directional resolution was limited by the step size used (1 µm). A topographic image of the area examined was produced and a histogram, showing the distribution of the profile departure density, was made. Several parameters were calculated of which three were chosen: root-mean-square deviation of the profile  $(R_n)$ , maximum peak height  $(R_n)$  and skewness of the profile  $(S_k)$ . In Fig. 20a we can see a surface profile of a handsheet made of beaten latewood fibres. The handsheet does not contain fines and the fibres can be discerned clearly. After the first calendering (Fig. 20b) the fibre surfaces are flattened, fibre widths increased and height differences diminished. With further calendering (Fig. 20c) the height differences are still further evened out. The corresponding distributions of the profile departure density can be seen in Fig. 21.

The profile distribution of the uncalendered latewood sample is very wide, but on calendering (Fig. 21) it becomes considerably narrower and after the second calendering the surface is extremely smooth. All the parameters studied decrease on calendering, especially the maximum peak height ( $R_p$ ).



Fig. 20 a) Surface of handsheet made of beaten latewood kraft fibres. The profile is taken from an area of  $125\mu$  m by  $125\mu$  m. b) Surface of the same sample as in Fig. 20a after first calendering. c) Surface of the same sample as in Fig. 20b after second calendering.

These pictures are also shown on page 850 (Vol 2.) in colour

759

b)

C)

a)



Fig. 21 The profile departure density distributions of the same sample as in Figs 20a-c. The left ends of the distributions have been shifted.



Fig. 22 Effect of slight calendering on the distribution of profile departure density.

### Effect of calendering on the microscopic structure of surfaces

The main effect of calendering is the z-directional compression of paper. The calendering pressure affects the paper surface unevenly and is more concentrated on the peaks than the troughs. This can be clearly seen in Fig. 22. The profile density distribution remains almost unchanged on slight calendering except at the right end of the distribution curve where the higher points have been compressed. As a result of the same uneven compression the profile distributions, without exception, result in negative skewness values, the values becoming more negative with further calendering. This implies that as the paper is subjected to additional calendering, the roughness to an increasing extent will be attributable to the effects exercised at the troughs.

The calendering causes changes also in the lateral direction. The changes in fibre widths are noticeable. This usually happens at crossing areas of two or more fibres. Also some displacement of fines and fibres in the lateral direction can take place. The amount of displacement seems to depend on the bonding of fibres and the length of free fibre segments.

# Comparison of the changes in the microroughness of earlywood and latewood sheets

The changes in the surface structure observed on the microscopic scale are not qualitatively different from those revealed by the macroscopic measurements. The earlywood samples also become smooth on the microscale and need less calendering than latewood samples and they reach an equivalent smoothness level at a lower apparent sheet density (Fig. 23). The addition of 15% TMP fines reduces the root-mean-square (RMS) microroughness as it reduces the PPS roughness. A correlation between microscopic parameters and macroscopic air leak results has been shown earlier ( $\underline{46}$ ). The more negative skewness values of latewood sheets imply that the smaller number of fibres in latewood sheets give rise to more and deeper troughs in the sheets. The skewness is, however, diminished by fines addition. On the whole, the fines addition does not make the surface optically rougher but offers a means of also improving the microscale smoothness.



Fig. 23 Effect of calendering on the root-mean-square deviation of profile measured by confocal laser scanning microscope.

### CONCLUSIONS

The quality and amount of fines is a potential control variable in papermaking. It seems to offer a possibility of simultaneously improving several properties of printing paper. By adding suitable fines the tensile strength, light scattering and smoothness of paper can be improved. The increasing drainage rate can be a limiting factor but it is also strongly dependent on the properties of the fibre fraction.

The type of fines or the composition of the fines mixture, controls the degree to which the optical, strength and smoothness properties are affected. The chemical composition and the specific surface area of the fines fraction seem to be the main factors characterizing the differences between the fines of mechanical and chemical pulps. The primary fines of a bleached kraft pulp are of distinctly lower quality than the fines created during beating. No large differences were found between fines produced at different stages of beating.

Properties of both earlywood and latewood kraft fibre networks can be improved by fines addition. An addition of kraft fines increases the tensile strength of earlywood sheets more than that of latewood sheets. The conformability and specific surface area of the fibres probably determine the response to fines addition and increased Campbell forces.

TMP fines are superior to kraft fines in improving the smoothness of uncalendered and calendered handsheets. TMP fines also improve the smoothness on the microscopic level. The skewness of the microscopic profile distribution is higher in latewood sheets than earlywood sheets. The addition of TMP fines reduces the skewness by reducing the severity and number of troughs.

Calendering usually augments the differences in paper properties evident between earlywood and latewood sheets. The latewood sheets demand more harsh calendering conditions which cause deterioration of most paper properties. The smoothness of latewood kraft sheets, however, can be improved to the level of pure earlywood fibre sheets by a high addition of TMP fines.

The addition of fines does not, however, diminish the importance of the fibre fraction. As the fines in most cases intensify the interaction between fibres, the effect of the fines depends on the qualities of the fibre fraction.

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# **Transcription of Discussion**

# EFFECT OF FINES ON THE PROPERTIES OF FIBRE NETWORKS

E Retaulainen

**ERRATA**: On p762 of this paper, 'drainage rate' should read 'drainage resistance'.

### Prof C T J Dodson, University of Toronto, Canada

It's always worrying for statistical geometry that these fines are lurking in the background with an accusing finger pointing at us, so it is very nice to have this documented so that we can refer to it. Two points, Elias, the first one is a question, but for the second point you might care to look at the conclusion you have on page 762. Your paper is very clear but that conclusion says the opposite of what you mean. When you say drainage rate I think you mean drainage resistance. But this is a trivial typographical error I think.

The technical question was, do you collect the through fraction so that you can estimate the fraction of fines retained?

### E Retulainen

Yes, white water recirculation was used so that over 90% of the fines were retained in the handsheets.

### J Unbehend, Weyerhaeuser Paper Company, USA

After 20 years of taking fines in and out of sheets and furnishes, I find it is nice to see them getting the attention that they have gotten in your work. One comment I would like to make is that we have found that the fines play an especially important role in TMP, and the same is true for fines from Kraft pulps. In work on TMP fines,

done in conjunction with Renarta Marton while at ESPRI in Syracuse, we found that there was distinct difference in performance if you were to sub-divide the fines into coarser and finer fractions. We even studied the unsettled portion of the fines fraction as well finding that the contribution to the consolidation of the sheet and the optical and physical properties were widely different for these fractions. We also saw this with Kraft fines, both hardwood and softwood, looking at different levels of refining. Most of the work that we did agrees with what you are doing and again, I congratulate you on your work and encourage others working in this area to continue. As Professor Dodson said, consider the fines and their contribution to paper properties from a reinforcement standpoint as well as their function in the overall structural network.

# E Retulainen

Thank you. I agree with what you said about this, regarding the quality of fines. I think that the fines fraction is not homogeneous and could be divided into at least three different fractions; a coarse fraction, a middle fraction and a fine fraction.

## Dr J R Parker, Messmer Instruments, UK

Most interesting. It doesn't surprise me that TMP fines act as fillers and they don't bond to the fibres, They are mobile and they can fill in the holes between the fibres, something that the kraft fibres might not be able to do but my point is this. I wonder what are the details of the microscopic technique you used for measurement. How did you support the sample when you measured its surface profile. Did you, as in a printing press, press the sample surface you were studying against something like a glass plate to simulate the printing plate, so you had a surface that was under the same conditions as it would be in the press nip. Or did you look at just the free surface in which case asperities on the surface would stick upwards and in fact bias the roughness histogram you have observed.

### E Retulainen

These were free surfaces not under pressure. The handsheet samples were placed on microscope slides and glass coverslips held firmly on top.

### **J** Parker

Sorry, you say that it was not under pressure.

### E Retulainen

Yes we calendered the handsheets and then studied them under the microscope looking at exactly the same areas as before calendering. It was not our intention to simulate the pressure that occurs at the surface of the paper when in contact with the printing plate. Our aim was to examine the changes in surface structure at the microscopical level that are induced by calendering treatments, and to see how different fibre networks respond to calendering.

### **J** Parker

Yes, may I explain. There has been quite a history of how to measure roughness. Initial measurements of roughness were made under fairly low pressure and it was found that this did not correspond to the results obtained on printing presses simply because the conditions of measurement did not correspond to the press nip. If you press the sample against a hard surface first so that it flattens as it would flatten against a printing plate and if you can observe the deviations between the printing plate and the surface of the sample, then you can see the surface which you have to attack and fill up with the ink in order to get a good print. That is my point.

### C Soremark, ASSI Kraftliner, Sweden

I have a question regards distinguishing between the primary and secondary fines. Did you start off with a dried pulp and could that be a part of the explanation for the difference in "efficiency" between the two types of fines you mentioned?

### E Retulainen

Yes, we used dry lap pulps but I understand that the difference between primary and secondary fines is similar also within neverdried pulps because the primary fines contain parenchyma cells and chunk like material which is not fibrillated. The secondary fines contain more fibrillated material and lamellae so I think that shape is the most important difference.

## Dr W Hewertson, CSIRO, Australia

In your list in table 1 most of the effects are positive other than the tear strength and of course drainage. Tear strength appears to be negative with increase of fines whereas later on in figure 5 you show an increase in bonding strength with fines in the Nordman bonding strength. Why do you think you get that strong reduction in tear strength with increasing fines?

### E Retulainen

I think that tear strength is strongly dependent on the long fibres, and depending where we are on the tear strength curve, and usually we are on the latter part where the tear strength goes down when we increase bonding. In this case we increase bonding when we add fines while at the same time we actually decrease the amount of long fibres.

### Prof E Back, Feedback Consulting E&E Back KB, Sweden

My first question was already answered in the discussion. The primary fines to a certain extent are made up of parenchyma and ray cells and have a little similarity to secondary fines which are fibrils and fibril containing fines in parenchyma cells might actually have a negative effect because of their olephilic material. My second question. In general papermaking you want to compare the addition of fines at equal drainage and your data if replotted might give some information on this. If you make a comparison at equal density, when density is varied, eg by wet pressing do fines still have a positive effect when added. Do you have any data in this respect?

### **E** Retulainen

The first thing I think there are differences in drainage between these two types of fines. Our paper was made in the sheet mould and if not more than 15% fines were added in most cases the drainage time did not increase much, in fact the increases were rather small. Only when we added 30% fines to the earlywood fractions did the drainage time go to 20 minutes or so. We have looked at strength against the density of handsheets and there is this normal trend that with increasing density, the tensile strength increases. There also seems to be some variation which might be due to this bond strength effect but this we have not thoroughly studied.